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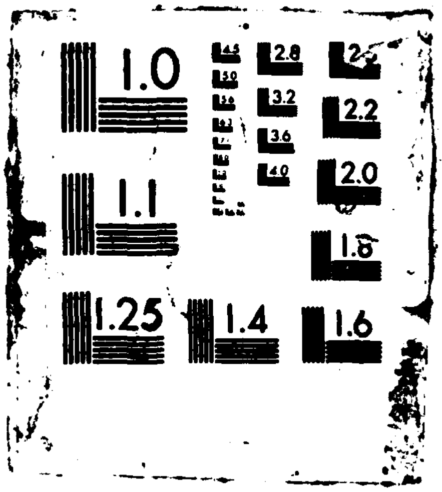
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SUBMICRON AEROSOL CHARACTERIZATION OF WATER BY A DIFFERENTIAL MOBILITY PARTICLE SIZER (U)

by

B. Kournikakis, A. Gunning, J. Fildes and J. Ho

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ABSTRACT

Submicron aerosols were generated from various water samples by a commercial generator. Submicron aerosol characteristics were determined using a differential mobility particle sizing instrument. Water purity was found to be directly related to submicron aerosol characteristics. It was shown that this new method of analyzing water is more sensitive than conventional methods. The units of measurement are more informative and relevant to modern science and industry.

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INTRODUCTION

In a preliminary study of the characterization of submicron aerosols of proteinaceous compounds, it was found that the solvent background spectrum was interfering with that of the sample used. It was concluded that impurities in the solvent, water in this case, was causing the interference (1). These impurities could include bacteria, suspended particles, gases, colloids or organic and inorganic compounds (2, 3).

Impurity levels in water are often measured in parts per million. Many applications of science and engineering, however, require water which is many times purer. In fact, the technology for obtaining water with impurities measured in parts per billion (0.01 ug/ml), was developed by the electronics industry to generate pure

water for silicon chip production (1). Similarly, many other fields of science, such as food production and medicine, require water of this purity (3, 4).

High purity water can be achieved by a variety of methods. These include settling, filtration, flocculation, distillation, ion-exchange, reverse osmosis, electrodialysis, degasification, and sterilization. The choice of one of these methods depends on the scale and purity level of the water required (5). Pure water has been shown to become recontaminated after one hour of storage or transport in metal, plastic or glass systems (6). Truly pure water is only produced when required (5).

Many methods of purity analysis are currently available and these can be either large or small scale detection systems (2). Methods range from gross qualitative studies such as color and clarity analysis to more sensitive quantitative conductivity studies (5) or analysis by high performance liquid chromatography coupled to UV detection (7). The choice of analysis method is dictated by the level of sensitivity and the scale of analysis required. However, these conventional methods suffer two deficiencies. Firstly, in many methods, conductivity measurements for example, as water purity increases, resolution and sensitivity decreases. Secondly, in processes that require quantitation of the particulate contamination, current measuring instruments do not provide a direct indication of the levels.

In this investigation, a new method of water characterization using aerosol production and analysis was devised. In many scientific and industrial applications, water is used as a solvent and is aerosolized or dried. By this process, any impurities in water will

ultimately appear as particles. These particles may cause undesirable effects in the final products. Thus, an expression of purity in terms of particle size distribution or numbers is more informative. The technique uses a TSI Differential Mobility Particle Sizer (DMPS) to characterize submicron aerosols within the range of 0.11 to 0.453 μm mass median diameter (MMD).

MATERIALS AND METHODS

The analysis and characterization of water samples was performed by analyzing the particle content of aerosols produced from a variety of water samples. The water samples studied were CFB Suffield tap water; single distilled water produced in copper still (Model A1016, Barnstead Co., Boston, MA, USA); triple glass distilled water produced by a Corning still (Model AG-11, Corning Glass Works, Corning, NY, USA) and ultrapure water produced by two systems, a Milli-Q/UF[R] system (Millipore Corp., Bedford, MA, USA) and a Nanopure[R] system (Sybron/Barnstead, Boston, MA, USA).

Submicron aerosols were generated as described below. The sample container (Pyrex, 1000 ml, Wheaton, Vineland, NJ) was rinsed several times with the water to be tested, then filled with 1 liter of the test sample. A filtered air supply (Model 3074, TSI, Inc.) operating at 241.3 kPa, pressurized a constant output atomizer (Model 3076, TSI, Inc.) to produce the aerosol. Subsequently the aerosol was passed through a diffusion dryer (Model 3062, TSI, Inc.) and a neutralizer (Model 3012, TSI, Inc.) before being drawn into a electrostatic classifier (EC) (Model 3071, TSI, Inc.). The classifier provided a step-wise selection of particle sizes for further analysis. The concentration of particles in each size range was determined by passage of the selected particles through a condensation nucleus counter (CNC)

(Model 3020, TSI, Inc.). This system was interfaced with an Apple IIe computer which provided the control for the stepwise regulation of the classifier voltage and data processing. Data output from the computer was expressed as concentration and size distribution of particles that were present in the samples.

Multiple sample mean comparisons data analysis was performed by the Student-Newman-Keuls test (SNK) (8). These authors also suggested Cochran's test for variance homogeniety. The SNK program, an IMSL subroutine (subroutine ASNKMC, International Mathematical and Statistical Libraries, Inc., Houston, Texas, 77036) was implemented to run on the Honeywell DPS 8 as was the Cochran's test. A program was written (Kakari Systems Ltd., Edmonton, Alberta) to extend the confidence range of the SNK analytical capability to cover alpha 0.05 to 0.4 in increments of 0.05. All programs were tested with standard data sets from IMSL as well as from Sokal and Rohlf. (8).

RESULTS AND DISCUSSIONS

Instrument blank was a critical consideration in measuring low concentrations of submicron aerosols. This was accomplished by performing the experiment with an empty sample vessel while all other instrumental parameters were maintained constant. By this procedure, absolutely no submicron particles were detected.

The aerosol particle size distribution spectrum generated from any given solution is a function of a number of parameters. One is the type of aerosol generator used to produce the aerosol. In this report, a submicron particle generator was employed. Another is the composition of the solutes and their concentrations. Tap water contains a complex mixture of solutes, mostly inorganic salts. Thus, an aerosol produced from it would most likely consist of a broad

spectrum of particle sizes in the submicron range. Figure 1 shows the size distribution of cold tap water and single distilled water. The particle mass median diameter (MMD) for tap water was about 0.05 μm while that for single distilled water was 0.03 μm. Analysis of means revealed that these were significantly different (Table 2).

Most evident was the significantly higher ($P=0.01$) concentration of particles derived from tap water compared to that of single distilled water (Fig. 1 and Table 1). This observation was consistent with the observation by conductivity measurements that tap water contained higher concentrations of dissolved materials. It was expected that water which contained even less solute would exhibit lower aerosol particle numbers. As shown in Figure 2, more sophisticated treatment of the water produced better quality water as depicted by lower particle numbers of significantly smaller MMD (Table 2). At the same time, the size distributions also tended to become narrower, perhaps indicating a less complex mixture of solutes. Initial improvements in each purification method corresponded to about one order of magnitude decrease in particulate aerosol content. However, quantitative particulate improvements between triple distilled and Milli-Q methods were comparatively less yet still significant (Table 2). This illustrates the sensitivity of the method.

During the time when these experiments were performed, two commercial ultrapure water systems (Milli-Q and Nanopure) based on similar technology were available. Each claimed that their system was superior to the other. The manufacturers' evidence to back up the claims were difficult to interpret. As shown in Figure 3, the particle size and number distributions were virtually the same. It was obvious, therefore, that there was no real difference between the quality of the water produced by these systems (Tables 1 and 2).

In working with ultrapure water from either system, it was noted that aging was a critical factor in water quality. Figure 4 shows results from measuring submicron aerosol content of aged ultrapure water (Milli-Q). The percentage increase in aerosol particle concentrations from a water sample stored in a glass vessel was plotted versus time. Most rapid deterioration was seen in the first 2.5 hours of storage. Thereafter, the rate of increase stabilized to a lower constant. This observation strongly implicates leaching from the glass vessel as a contributing factor to the deterioration in water quality.

The current state of ultrapure water technology as exemplified by the two commercial instruments tested in these experiments, provide an adequate product for most scientific research. Water from these devices register about 0.15 microsiemens (μS) on the conductivity scale (SI units) (results not shown). This is the lowest limit of detectability for a modern day conductivity meter with new electrodes. On the other hand, the method as described here, although complex, offers at least two to three orders of magnitude better performance in detectability and resolution. The results obtained from replicate samplings of ultrapure water showed extremely low variance (Table 1). This suggests a certain degree of dependability in the method proposed here.

CONCLUSIONS

1. Impurities in water can be measured as total submicron aerosol particulate content, as well as particle size distribution.

2. Purifications methods reduce impurities which is reflected as reduction in submicron aerosol particle content.

3. Plots of particle size versus number concentration provide a very good graphical description of water quality.

4. Water quality is directly related to submicron aerosol particle concentration, MMD and spectral bandwidth.

5. The method described here represents a significant advance in that it produces an increased sensitivity in the measurement of water quality.

6. The unit of measurement (total particles) is directly relevant to several major industries (eg., microelectronics, pharmaceuticals, etc.).

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TABLE 1

COMPARISON OF WATER TYPES

SIGNIFICANCE LEVEL: .01

SAMPLE/ /MEAN	MILL 4.220	NANO 4.326	DIS3 4.598	DIST 5.068	TAP 6.261*
MILL:4.220	--	ND	SD	SD	SD
NANO:4.326	ND	--	SD	SD	SD
DIS3:4.598	SD	SD	--	SD	SD
DIST:5.068	SD	SD	SD	--	SD
TAP :6.261	SD	SD	SD	SD	--

* LOG 10 TOTAL PARTICLES/CC

VARIANCE HOMOGENEITY

TAP --- MEAN = 6.260975; VARIANCE = .00641
DIST --- MEAN = 5.068350; VARIANCE = .00152
DIS3 --- MEAN = 4.597684; VARIANCE = .00739
NANO --- MEAN = 4.326349; VARIANCE = .00401

VARIANCE HETEROGENEITY

MILL --- MEAN = 4.219836; VARIANCE = .02017

LEGEND: MILL = Milli-Q Water
NANO = Nanopure Water
DIS3 = Triple Distilled Water
DIST = Copper Distilled Water
TAP = Cold Tap Water
SD = Significant Difference
ND = No Difference

TABLE 2

COMPARISON OF GEOMETRIC MEANS

SIGNIFICANCE LEVEL: .01

SAMPLE/ /MEAN	MILL .0162	NANO .0168	DIS3 .0200	DIST .0280	TAP .0516
MILL:.0162	--	ND	SD	SD	SD
NANO:.0168	ND	--	SD	SD	SD
DIS3:.0200	SD	SD	--	SD	SD
DIST:.0280	SD	SD	SD	--	SD
TAP :.0516	SD	SD	SD	SD	--

VARIANCE WAS TOO LOW TO MEASURE

LEGEND: MILL = Milli-Q water
NANO = Nanopure Water
DIS3 = Triple Distilled Water
DIST = Copper Distilled Water
TAP = Cold Tap Water
SD = Significant Difference
ND = No Difference
UNIT = Micrometers

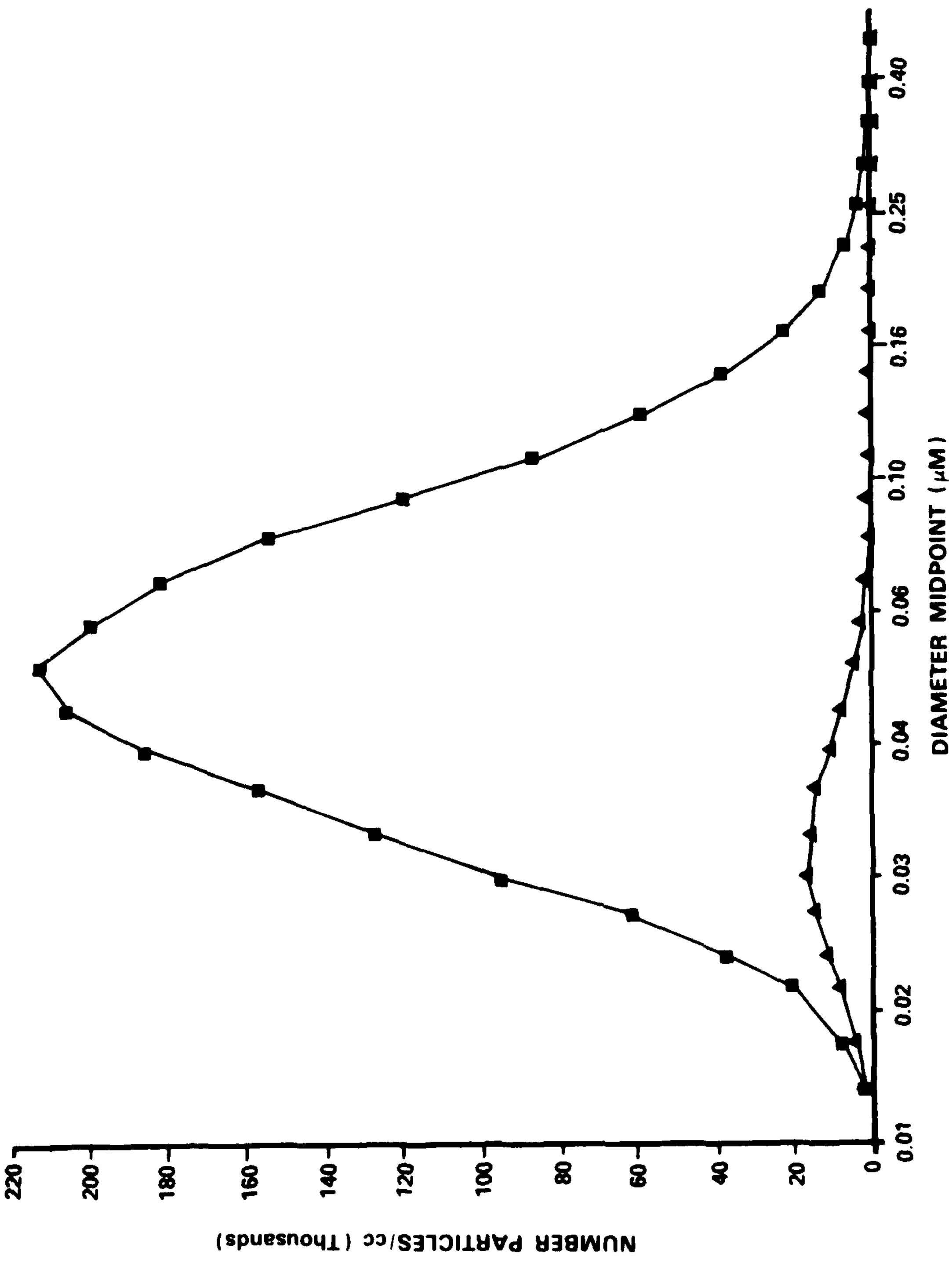

Figure 1

Particle Size Distribution of CFB Suffield Cold Tap Water (■) Compared to Single Distilled Water from a Barnstead A1016 Still (▲).

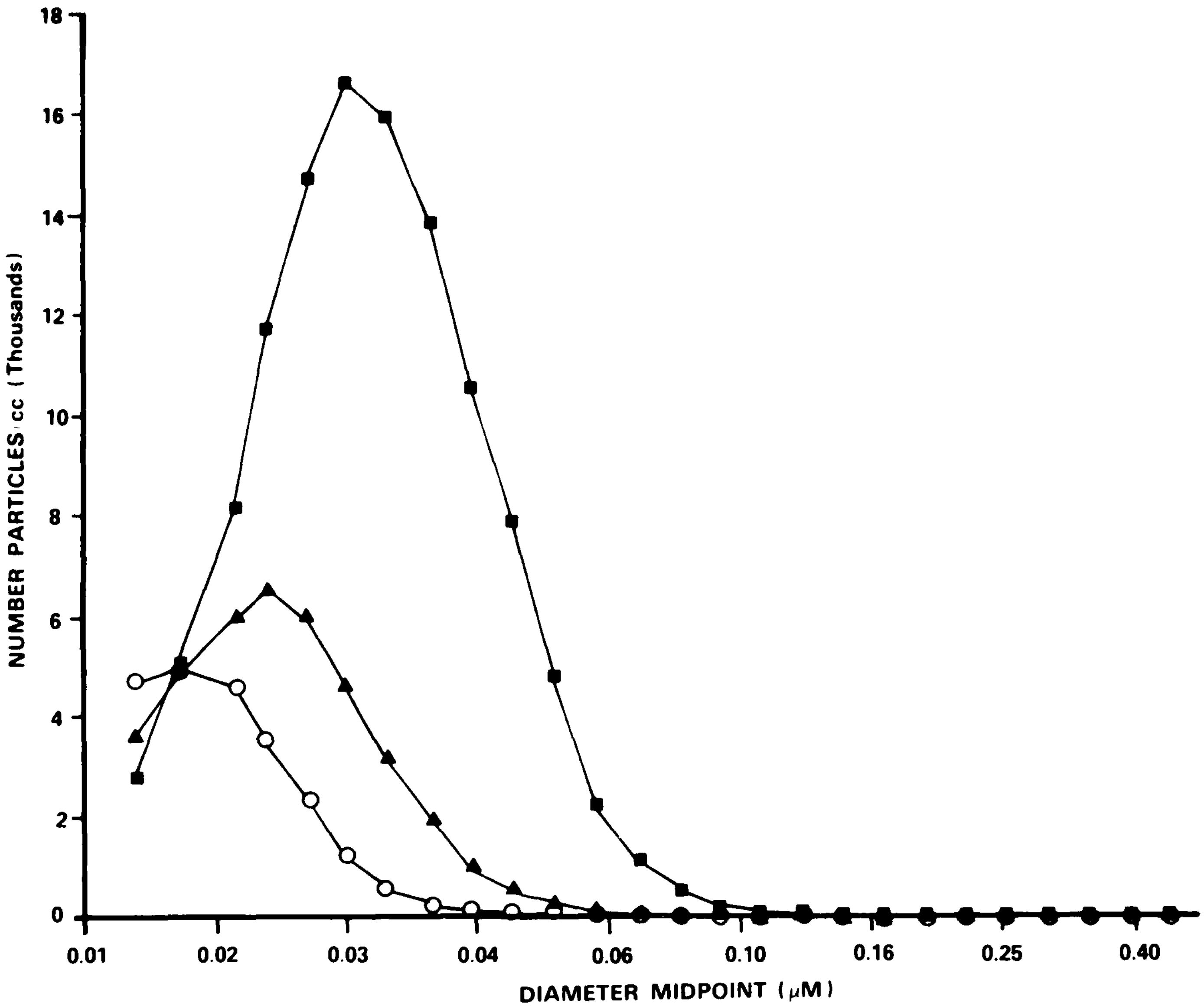

Figure 2

Comparison of Particle Size Distributions from Single Distilled Water from a Barnstead A1016 Still (■), Triple Glass Distilled Water from a Corning Model AG-11 Still (▲) and Ultrapure Water from a Millipore Milli-Q U/F System (○).

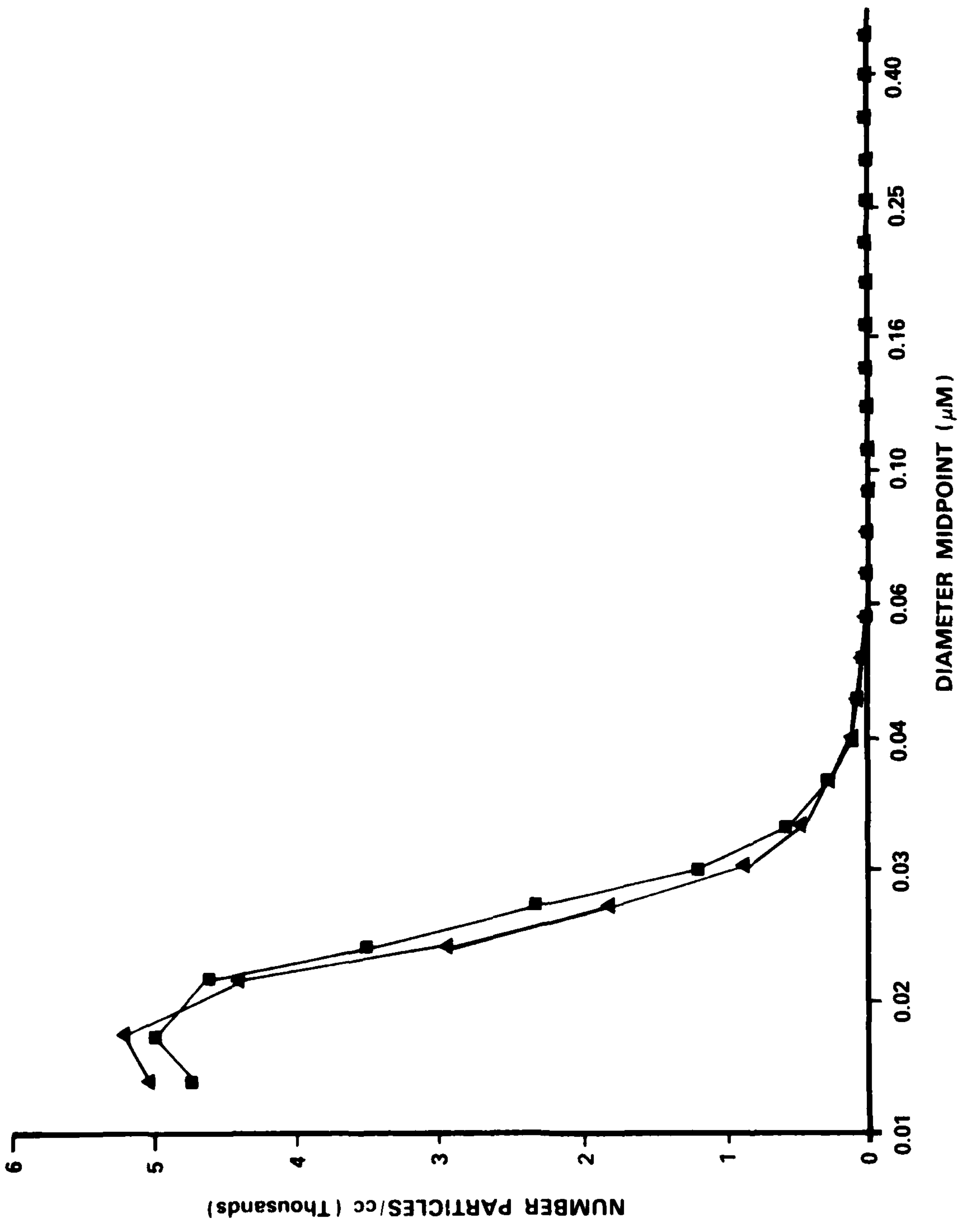

Figure 3

Particle Size Distributions of Ultrapure Waters Produced by the Millipore Milli-Q U/F System (■) and the Sybron/Barnstead Nanopure System (▲).

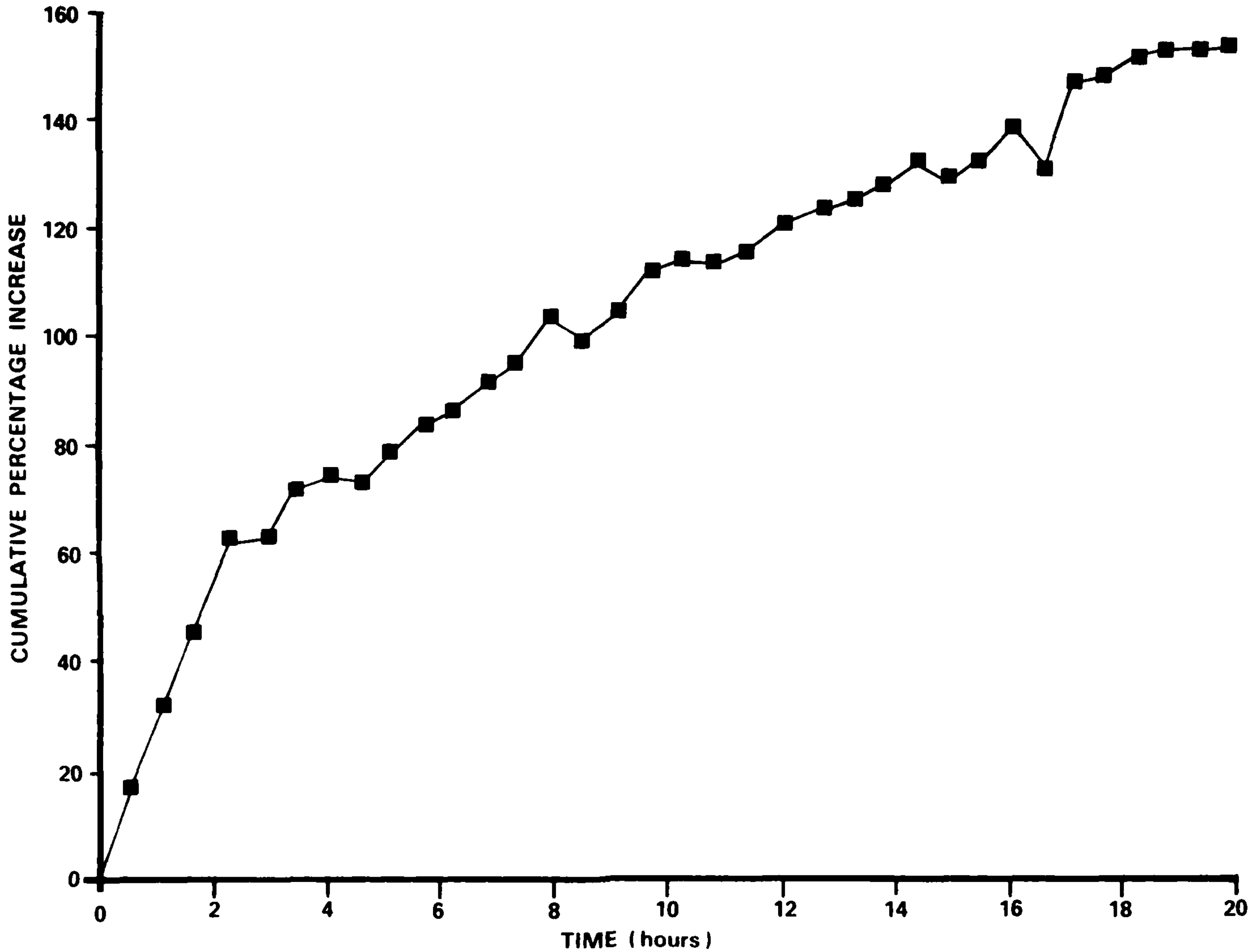

Figure 4

Cumulative Percentage Increase in Total Particle Counts in Ultrapure Water (Milli-Q) Aged for 20 hours in a 1 liter Wheaton Bottle.

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